

**HEAT-SEALABLE MULTILAYER WHITE OPAQUE FILM**

The present application claims the benefit of U.S. provisional application Serial No. 06/159,205 filed October 13, 1999.

**BACKGROUND OF THE INVENTION**

The present invention relates to multilayer plastic films and, more particularly, to heat sealable multilayer white opaque films suitable for packaging of heat-sensitive items.

Plastic films are currently used in many food packaging operations. To be commercially viable, these plastic films must be economically priced, be compatible with modern high speed packaging machinery and methods, and be suitable for the particular packaging application.

It will be appreciated by those skilled in the art that certain packaging applications, e.g., packaging of heat-sensitive items such as frozen novelties, including ice cream bars and ice cream sandwiches, introduce certain specific design criteria into the packaging operation. These specific design criteria include sealability at low temperature and with minimum applied pressure, a distinctive pleasing appearance with at least one printable surface, and compatibility with high speed packaging, machinery and methods. To date, the prior art has been unable to provide a film exhibiting the aforementioned design criteria suitable for packaging heat-sensitive items such as frozen novelties, including ice cream bars and ice cream sandwiches.

There is therefore a need in the art for a heat sealable multilayer white opaque film which is sealable at low temperature and with a minimum of applied pressure, provides a distinctive pleasing appearance with at least one printable surface, and is compatible with high speed packaging machinery and methods.

### SUMMARY OF THE INVENTION

The present invention, which addresses the needs of the prior art, relates to a heat-sealable multilayer white opaque plastic film which includes a cavitated polypropylene core layer having a first and a second surface. A top tie layer formed of polypropylene and incorporating a whitening agent is positioned adjacent to the first surface of the core layer. A top skin layer of polypropylene, or a polyolefin terpolymer, containing an antiblock agent, overlays the top tie layer. The film also has a polypropylene bottom tie layer positioned adjacent to the second surface of the core layer. A bottom skin layer of a polyolefin terpolymer and one or more antiblock agents or antiblock slip agents is positioned adjacent to the bottom tie layer.

In a first embodiment, the present invention provides a plastic film that is heat sealable on one side. The top (non-sealable) skin layer of the film is formed from polypropylene. The cavitating agent incorporated into the polypropylene core layer is a polybutylene terephthalate polymer. The top and bottom tie layers are formed from polypropylene and the polyolefin polymer of the bottom skin layer is a heat sealable ethylene-propylene-butylene terpolymer. Silicone oil is used as an antiblock agent and the antiblock slip agent is a crosslinked silicone.

In a second embodiment, the present invention provides a plastic film that is heat sealable on both sides. The top skin layer is an ethylene-propylene-butylene terpolymer containing  $\text{SiO}_2$  and an antiblock agent. The core layer contains a polybutylene terephthalate cavitating agent, a phosphite antioxidant, and a fluoropolymer as the anti-condensing agent. The polyolefin polymer of the bottom skin layer is an ethylene-propylene-butylene terpolymer, with silicone oil as the antiblock agent, and a crosslinked silicone as the antiblock slip agent.

The present invention also relates to a method of packaging a frozen novelty. The method includes the step of providing a frozen ice cream preparation. The method further includes the additional step of enclosing the ice cream preparation in a heat sealable white opaque multilayer plastic film. Finally,

the method includes the step of sealing the film to enclose the frozen ice cream preparation.

Thus, the present invention provides a packaging film which is sealable at a low temperature, and which is suitable for use with heat-sensitive items such as frozen novelties, including ice cream bars and ice cream sandwiches. Furthermore, the new films are compatible with modern high speed packaging machinery and methods, and are receptive to printing and labeling for marketing appeal.

These enhanced properties are achieved because the film of the present invention provides certain desirable characteristics including reduced plate out (wear of machine surfaces due to scouring and abrasion by exposed film components, especially hard additives such as titanium dioxide,  $\text{TiO}_2$ ), and a consistent low coefficient of friction (COF), good hot slip properties and improved hot tack and z-tear resistance for packaging. As a result, the film seals with a minimum of applied heat or pressure and still has a pleasing appearance, with at least one printable surface.

### **DETAILED DESCRIPTION OF THE INVENTION**

The present invention provides a multilayer white opaque plastic film heat-sealable on one or two sides, with improved hot tack and z-tear resistance characteristics; reduced plate out; a lower and consistent coefficient of friction (COF); good hot slip properties; and with a sealant layer that has a distinctive pleasing appearance. At least one exposed surface of the film is suitable for receiving an image which may be printed or affixed.

The plastic film of the present invention includes at least five layers. The core layer is the central layer of the five layer film structure. On each surface of the core layer is a tie layer. An outer skin layer is present on each side of the multilayer structure.

Each layer itself is formed from one or more polyolefin polymer compositions. Suitable polyolefin polymers include for example, polypropylene (PP), ethylene-propylene copolymers (EP), and ethylene-propylene-butylene

terpolymers (EPB). It will be appreciated that skin layers formed from EP copolymers or EPB terpolymers typically exhibit heat-sealing properties. Thus, a multilayer film having one skin layer formed from heat sealable polymers is heat-sealable on one side, whereas a multilayer film having both skin layers of a heat sealable polymer is heat-sealable on two sides.

The multilayer polyolefin films of the present invention are opaque, white films. The opacity and whiteness characteristics are due to the presence of whitening agents, particles and cavitation in one or more layers of the film.

Preferably, the core layer of the multilayer film is rendered opaque as a result of cavitations within the layer. This cavitation is accomplished by adding an amount of a cavitating agent to the core layer prior to stretching of the film. When the multilayer film is subsequently stretched, the cavitating agent produces voids in the core layer which engender a characteristic opacity to the film. The cavitating agent may be any substance which is harder than the polyolefin of the layer to which it is added. Suitable cavitating agents include any polymer which is harder than the matrix polymer, such as, for example polybutylene terephthalate (PBT) in a polypropylene (PP) matrix.

The whitening agent or agents, which are incorporated into at least one layer of the film, also contribute to the whiteness and opacity of the films of the present invention. At least one of the tie layers of the film comprises a whitening agent. Alternatively, the whitening agent may be present in two tie layers. Suitable whitening agents include but are not limited to white pigments, such as for example  $\text{TiO}_2$ ,  $\text{CaCO}_3$ ,  $\text{BaSO}_4$ ,  $\text{ZnS}$ ,  $\text{MgCO}_3$ , clay, talc, kaolin, or any other highly reflective white compound. In a preferred embodiment, the whitening agent is  $\text{TiO}_2$ . In an even more preferred embodiment, the whitening agent is the  $\text{TiO}_2$  product, Millenium RCL4<sup>®</sup>

Other additives and agents may be suitable for incorporation into one or more layers of the films of the present invention. Additives may be selected from any class of additives, including for example, antioxidants, anti-condensing agents, slip agents, pigments, fillers, foaming agents, flame retardants, photodegradable agents, UV sensitizers or UV blocking agents, crosslinking

agents, silicon compounds (e.g.  $\text{SiO}_2$ ) and anti-block agents to name but a few of the many known additives.

Anti-oxidants suitable for incorporation in the films of the present invention may be from any class of anti-oxidant, such as a phosphite, for example Ultrinox<sup>®</sup> 626. The anti-condensing agent may be any anti-condensing agent, for example a fluoropolymer, such as for instance Dyneon<sup>®</sup> fluoropolymer FX9613. The antiblock agents suitable for use with the films of the present invention may be any antiblock agent, including those which function as antiblock, slip agents, for example a methyl acrylate such as Epostar<sup>®</sup> MA 1002 (Nippon Shokubai, Osaka, Japan).

The silicon compounds include various forms of  $\text{SiO}_2$ , which may be for example, in the form of coated or uncoated silica including for example, Sylobloc<sup>®</sup> 44 and Sylobloc<sup>®</sup> 45, respectively, supplied by W.R. Grace; alternatively, the silicon compound may be, for example a silicone oil. The silicone oil may be any silicone oil, for example, SH<sup>®</sup> 200. Many crosslinked silicone compounds are commercially available and are useful for incorporation into the films of the present invention. These include, for example, the preferred crosslinked siloxane compound Tospearl<sup>®</sup> manufactured by Toshiba Silicone, Tokyo, Japan.

One or both of the exposed surfaces of the film of the present invention may be treated to provide the film of the present invention with further useful properties and functionalities. These include for example corona treatment and treatments that enhance receptivity for printing, especially for good compatibility with water based inks.

Optimum film characteristics for machining include a low coefficient of friction, COF (good slip properties) and low block, i.e. the film surfaces should not stick together and should not interfere with rolling and packing. These characteristics are imparted by the slip agents and the antiblock agents, respectively, of the films of the present invention.

In particular, the present invention provides a heat-sealable multilayer white opaque plastic film in which the core layer is cavitaded and is formed from polypropylene. The film has a top tie layer of polypropylene and a whitening

agent. This top tie is positioned adjacent to the first surface of the core layer. A top skin layer of polypropylene or a polyolefin terpolymer is positioned adjacent to the top tie layer. The top skin layer also contains  $\text{SiO}_2$  and an antiblock agent. A bottom tie layer formed from polypropylene is positioned adjacent to the second surface of the core layer. A bottom skin layer formed from a polyolefin terpolymer is positioned adjacent to the bottom tie layer and contains  $\text{SiO}_2$ . The bottom skin layer may further contain one or more other antiblock agents or antiblock slip agents.

The core layer preferably includes from about 7% to about 9% cavitating agent, which may be any hard small particle compound, preferably PBT optimally present at 8%; from about 500ppm to about 700ppm of an antioxidant, preferably a phosphite, optimally present at 600ppm; and from about 200ppm to about 400ppm of an anti-condensing agent, preferably a fluoropolymer and optimally the fluoropolymer is present at 300ppm.

The top tie layer preferably include up to 10%  $\text{TiO}_2$ , preferably up to 8%, more preferably up to 6%, and optimally about 4%  $\text{TiO}_2$ .

The top skin layer preferably includes from about 0.1% to about 0.5%  $\text{SiO}_2$ , preferably from about 0.15% to about 0.4%  $\text{SiO}_2$ , more preferably from about 0.2% to about 0.3%  $\text{SiO}_2$ , and optimally about 0.23%  $\text{SiO}_2$ ; and from about 0.1% to 0.5% of the antiblock agent, preferably from about 0.2% to about 0.4%, more preferably from about 0.15% to about 0.3% of the antiblock agent, and optimally about 0.2% of the antiblock agent.

The bottom skin layer preferably includes: EPB terpolymer and from about 0.6% to 2.4% of an antiblock agent which may be any antiblock agent, for example a silicone oil, preferably the antiblock agent comprises from about 0.9% to 1.8% of the bottom skin layer (optimally 1.2%); from about .05% to about .15%  $\text{SiO}_2$  (optimally 0.1%  $\text{SiO}_2$ ) from about 0.15% to about 0.3% of an antiblock slip agent, which may be any antiblock slip agent, such as for example a crosslinked silicone (optimally comprising 0.23% of the layer).

In a yet further embodiment of the first aspect of the invention the total film thickness of the heat-sealable multilayer white opaque plastic film is about 1mil and the top skin layer constitutes about 2.5% of the total film thickness; the

top tie layer forms about 15% of the total film thickness, the core layer accounts for about 63% of the total film thickness, the bottom tie layer forms about 15% of the total film thickness, and the bottom skin layer constitutes about 4% of the total film thickness.

In a particularly favored embodiment, the five layer white opaque plastic film of the present invention is heat sealable on one side and has the following composition: the top skin layer is corona treated and is formed from polypropylene and 0.23% Sylobloc<sup>®</sup> 45 (W.R. Grace, New York, NY) and 0.2% Epostar<sup>®</sup> MA1002; the top tie layer is formed from polypropylene and 4% Millenium RCL4<sup>®</sup>; the core layer is formed from polypropylene and includes 8% BPT, 600ppm Ultrinox<sup>®</sup> 626 (Borg-Warner Chemicals Inc., Parkersburg, VA), and 300ppm of the fluoroplastic, Dyneon<sup>®</sup> fluoropolymer FX9613 (3M, St. Paul, MN); the bottom tie layer is also formed from polypropylene, and the bottom skin layer is formed from the EPB terpolymer, Chisso<sup>®</sup> 7753 and includes 1.2% SH200, 0.1% Sylobloc<sup>®</sup> 44 (W.R. Grace, New York, NY) and 0.23% Tospearl<sup>®</sup> 130 (Toshiba Silicone, Tokyo, Japan).

In another particularly favored embodiment, the five layer white opaque plastic film of the present invention is heat-sealable on two sides and has the following composition: the top skin layer is corona-treated and is formed from an EPB terpolymer, Chisso 7300 and includes 0.23% SiO<sub>2</sub> as Sylobloc<sup>®</sup> 45, 0.2% antiblock agent as Epostar<sup>®</sup> MA1002 (Cross-linked polymethacrylate, with a 2-3um average particle size; a refractive index of 1.49, and a specific gravity of 1.2), the top tie layer includes 4% TiO<sub>2</sub> as Millenium RCL4<sup>®</sup>; the core layer includes polypropylene and 8% of a cavitating agent, which is PBT; 600ppm of an antioxidant phosphite which is Ultrinox<sup>®</sup> 626 and 300ppm of an anti-condensing agent which is the Dyneon<sup>®</sup> fluoropolymer FX9613; the bottom skin layer is formed from the EPB terpolymer, Chisso<sup>®</sup> 7753 (Chisso Corp., Osaka, Japan); and includes the antiblock agents, silicone oil, SH 200<sup>®</sup> at 1.2%, uncoated silica, Sylobloc<sup>®</sup> 44 (W.R. Grace, New York, NY) at 0.1% and 0.23% of the crosslinked silicone, Tospearl<sup>®</sup> 130 (Toshiba Silicone, Tokyo, Japan).

**EXAMPLES****Example 1 (Comparative Example)**

A three layer, one side heat sealable plastic film of the following composition was manufactured and provided for comparative purposes:

-----This Surface Treated-----

Layer 1	Exxon <sup>®</sup> 4612 PP + 4% Millenium RCL4 <sup>®</sup> TiO <sub>2</sub>	25 ga
Core	PP homopolymer of high stereo-regularity, e.g. Exxon <sup>®</sup> 4612 + 8% PBT cavitating agent: Ticona Celanese <sup>®</sup> 1300A or equivalent	65 ga
Layer 2	EPB terpolymer + 0.23% Sylobloc <sup>®</sup> 44 + 1.25% silicone fluid: 30,000 centistokes	8 ga

The layers were combined in the melt, quenched, reheated, and stretched to 5.3X in the machine direction. Subsequently, the sheet was reheated and stretched about 8-10X in a tenter frame. Skin 1 was corona treated to about 40 dynes and wound in a mill roll form.

**Example 2**

The following one side heat-sealable, five layer film structure was produced according to the process of Example 1.

-----This Surface Treated-----

Layer A	PP + 0.23% Sylobloc <sup>®</sup> 45 + 0.2% Epostar <sup>®</sup> MA 1002	2-3 ga
Layer B	PP (Exxon <sup>®</sup> 4612) + TiO <sub>2</sub> (4% Millenium RCL4 <sup>®</sup> )	15 ga
Layer C	PP homopolymer of high stereo-regularity, such as Exxon <sup>®</sup> 4612 + 8% PBT cavitating agent (Ticona Celanese <sup>®</sup> 1300A or equivalent)	61 ga
Layer D	PP (isotactic homopolymer)	15 ga
Layer E	EPB terpolymer (with DSC melting point of 122.5°C) + 0.10% Sylobloc <sup>®</sup> 44 + 0.1% Tospearl <sup>®</sup> T130 + 1.25% silicone fluid (30,000 centistokes)	4 ga



**Example 3**

The following two side heat-sealable, five layer film structure was produced according to the process of Example 1.

-----This Surface Treated-----

Layer A	EPB terpolymer (with DSC melting point of 137°C) + 0.23% Sylobloc® 45 + 0.2% Epostar® MA 1002	2-3 ga
Layer B	PP (Exxon® 4612) + TiO <sub>2</sub> (4% Millenium RCL4®)	15 ga
Layer C	PP homopolymer of high stereo-regularity, such as Exxon® 4612. 8% PBT cavitating agent (Ticona Celanese® 1300A or equivalent)	61 ga
Layer D	PP (isotactic homopolymer)	15 ga
Layer E	EPB terpolymer + 0.10% Sylobloc® 44 + 0.1% Tospearl® T130 + 1.25% silicone fluid (30,000 centistokes)	4 ga

The films of Examples 2 and 3 have the following advantages over the film of comparative Example 1:

- (a) The layer containing TiO<sub>2</sub> may be reduced from 25 ga in Layer 1 of Example 1 to 15 ga in Layer B of Examples 2 and 3, representing a 40% reduction in materials cost for this layer.
- (b) The sealable layer may be reduced from 8 ga in Layer 3 of Example 1 to 4 ga of Layer E of Examples 2 and 3, representing a 50% lower material cost.

The film of Example 3 is heat sealable on two sides, with a somewhat peelable seal, and is suitable for ice cream sandwich applications.

To simulate film performance in manufacturing, the following tests were run:

1. COF (Coefficient of friction).
2. Hot slip (Film on metal at high temperatures).
3. TiO<sub>2</sub> plate out testing ink adhesion monitoring on a Chestnut press (Flexo printing machine).
4. Fuji HFFS (Horizontal form, fill and seal): Testing for machinability.

- Hayssen VFFS (Vertical form, fill and seal):** Laminating a standard film (50LBW) to the test film sample(s) and testing hot tack with 16oz kidney beans as the test load.

Table 1 lists the performance and properties of these films.

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**TABLE 1: Film Properties and Performance**

Example	COF		Treated side hot slip		TiO <sub>2</sub>	Ink Adhesion		Fuji HFFS		Hayssen VFFS	
	T/T	U/U	250°F	275°F	Plate-out	Solvent	Water	Machinability	Seal @290°F (gm/in)	Hot tack	Seal @270°F (gm/in)
1	0.34	0.41	0.59	0.6	Failed	Passed	OK	Passed	405	Creep 280-310°F	645
2	0.26	0.23	0.63	0.69	Passed	Passed	OK	Passed	695	No Creep	945
3	0.25	0.23	0.92	1.48	Passed	Passed	Passed	Passed	695	No Creep	945
For TiO <sub>2</sub>											
Failed = Can wipe TiO <sub>2</sub> off film surface						Passed = > 95% ink adhesion					
Passed = Cannot wipe TiO <sub>2</sub> off film surface						OK = 50 to 95% ink adhesion					

As can be seen from Table 1, the films of Examples 2 and 3 provide a significantly lower and more consistent COF than the film of Example 1. The films of Example 1 and Example 2 provide good hot slip properties and eliminate plate out of the  $\text{TiO}_2$ . The films of Example 2 provide good ink adhesion properties, while the films of Example 3 provide improved ink adhesion properties. The films of Example 2 and 3 provide good machineability. Finally, the films of Example 2 and 3 provide improved hot tack, and have improved z-tear performance (without creep), which is important for VFFS (Vertical form, fill and seal) applications.